Technobius Physics, 2025, 3(2), 0031, DOI: https://doi.org/10.54355/tbusphys/3.2.2025.0031

Technobius Physics

https://technobiusphysics.kz/

e-ISSN
3007-0147

Article

Structural analysis of NaCl and CsCl via Debye–Scherrer powder diffraction

Marry Brewer*, Almas Sheriyev

Department of Materials Science, Sheffield Hallam University, Sheffield, United Kingdom
*Correspondence: b248967468@gmail.com

Abstract. This study investigates the crystal structures of sodium chloride and caesium chloride through powder X-ray diffraction using the Debye-Scherrer method. The primary objective was to determine lattice types, calculate lattice constants, and estimate the number of atoms per unit cell based on experimental diffraction ring data. Powdered samples were exposed to X-rays, and the resulting ring patterns were analyzed to assign Miller indices and derive interplanar spacings. The evaluation of ring positions and intensities revealed that sodium chloride crystallizes in a face-centered cubic structure, while caesium chloride adopts a body-centered cubic arrangement. The calculated lattice constants were 562.0 pm for sodium chloride and 409.6 pm for caesium chloride, both in good agreement with standard reference values. Additionally, the number of atoms per unit cell was determined to be approximately four for sodium chloride and two for caesium chloride, consistent with their respective crystal symmetries. The study confirms the reliability of basic X-ray diffraction techniques for structural identification in ionic solids and highlights distinct diffraction trends corresponding to different cubic symmetries. These findings reinforce the effectiveness of the Debye-Scherrer approach in crystallographic education and rapid phase analysis, while also identifying opportunities for enhanced resolution through extended exposure or high-intensity sources.

Keywords: X-ray diffraction, Debye-Scherrer method, crystal structure, sodium chloride, caesium chloride, lattice constant.

1. Introduction

Crystalline solids possess periodic atomic arrangements that define their physical properties and play a central role across materials science, solid-state physics, and crystallography. X-ray diffraction (XRD) is a powerful technique to probe these arrangements, and the Debye–Scherrer method, which analyzes powder samples, is particularly useful for determining structural properties such as lattice parameters and plane orientation through diffraction rings [1], [2], [3].

In cubic ionic crystals, determining whether a material adopts a face-centered cubic (fcc) or body-centered cubic (bcc) lattice is vital for understanding ionic packing density, stability, and phase behavior. Sodium chloride (NaCl) crystallizes in an fcc (rock-salt) structure, while caesium chloride (CsCl) exhibits a B2 (simple cubic with centered cation) structure often referred to as bcc-like [4], [5]. Distinguishing between these lattices using Debye–Scherrer data provides both educational and practical insights, especially when reflections overlap.

Nevertheless, contemporary challenges in Debye–Scherrer usage persist. Studies such as [6] have enhanced resolution to distinguish polymorphic alkali halides but reported ambiguities in indexing weaker peaks. [7] examined CsCl under thermal cycling, highlighting the influence of peak broadening on lattice constant determination. Automated indexing frameworks like those proposed by [8] have improved assignment reliability, yet they often fail when limited to low-angle data. Additionally, pressure-induced transitions (e.g., NaCl→CsCl phase change) underscore the method's sensitivity but also its complexity under dynamic conditions [9]. While advanced sources like synchrotron radiation and machine-learning platforms offer high precision [10], [11], conventional laboratory Debye–Scherrer setups remain more accessible and cost-effective [12].

A significant gap remains: there's no widely adopted, stepwise, reproducible protocol for accurately identifying fcc and bcc structures from basic powder camera data and simple measurements. Past research often assumes ideal peak clarity or requires advanced tools, limiting practical application in teaching or routine materials evaluation.

We hypothesize that by systematically analyzing ring diameters, $sin^2(\theta)$ quotients, lattice constants, and unit-cell densities from Debye–Scherrer patterns of NaCl and CsCl, one can reliably distinguish fcc from bcc structures using only standard laboratory equipment.

Therefore, this study aims to:

1. Obtain diffraction data from powdered NaCl and CsCl samples using the Debye–Scherrer method.
2. Assign Miller indices, compute lattice constants, and estimate the number of atoms per unit cell.
3. Demonstrate that these analyses can reveal crystal structure reliably and reproducibly in educational or QC laboratory settings.

This work provides a clear, physically grounded workflow that reinforces crystallographic fundamentals and highlights the practical utility and limitations of simple XRD techniques.

2. Methods

Materials and Sample Preparation

Sodium chloride (NaCl, 99.5% purity, 250 g) was used as the crystalline sample for X-ray structural analysis. To prepare samples of suitable optical thickness, the NaCl was first pulverized using an agate mortar. The powdered sample was then loaded into a holder created from punched standard printer paper (2–3 layers) sealed with transparent adhesive tape to form a shallow containment cavity. This setup allowed precise control of sample thickness in the range of 0.2–0.4 mm (Figure 1).

a) sample preparation b) adding the powder c) making the surface smoother d) attaching the diaphragm tube

Figure 1 – The process of NaCl samples preparation

Thickness within this range was critical to balance between sufficient edge absorption visibility and avoidance of excessive beam attenuation. Prepared samples were stored in sealed containers with silica gel to prevent moisture absorption, consistent with standard procedures for hygroscopic materials.

Experimental Setup

All measurements were performed using the XR 4.0 expert X-ray analysis system (Gulmay Ltd., UK), which included the XR 4.0 structural analysis upgrade set, XR 4.0 Plug-in Cu X-ray tube, and integrated control interface. The X-ray tube was operated at maximum capacity, with an anode voltage of 35 kV and an anode current of 1 mA, as per manufacturer guidelines.

Prior to initiating the experiment, the goniometer was removed from the internal chamber. A 1 mm-diameter diaphragm tube was installed in the beam outlet of the plug-in unit to limit the divergence of the primary beam. The X-ray-sensitive film was loaded into a light-proof film holder, which was positioned 35 mm from the NaCl crystal surface on the optical bench. The film plane was carefully aligned to remain parallel to the crystal face to ensure uniform angular resolution across the diffraction pattern. This fixed geometry was essential for consistent ring projection and accurate post-exposure evaluation.

The exposure was conducted under total darkness for a preset duration of 2.5 hours. Time tracking was performed automatically by the XR 4.0 software interface, which displayed a countdown timer and bar-graph progress. Post-exposure, the film was developed according to standard photographic chemical processing guidelines provided by the film manufacturer. The sequence included developer immersion, water rinsing, a 10-minute fixative bath, a final rewash, and air-drying.

Data Acquisition and Processing

The exposed films were analyzed using manual ring measurements, converted to diffraction angles using established geometric relationships for flat film geometry. The data acquisition was facilitated by the XR 4.0 software suite, which also recorded system parameters, exposure conditions, and environmental data (Figure 2).

a) selecting parameters b) starting a program

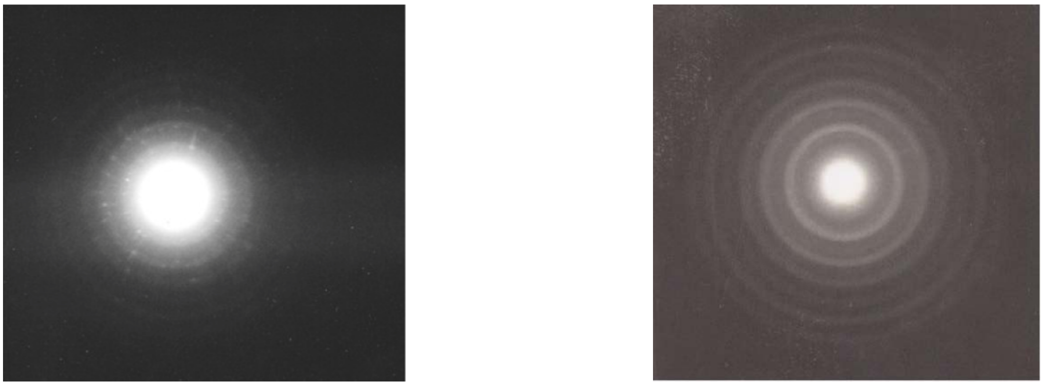
Figure 2 – X–Ray equipped special softwear

Uncertainty propagation was applied to angular measurements using standard error formulas, and regression analysis was carried out using MATLAB R2023a, applying nonlinear least squares fitting routines to extract structural parameters.

3. Results and Discussion

The structural characteristics of sodium chloride (NaCl) and caesium chloride (CsCl) were analyzed using Debye-Scherrer powder diffraction. The results are presented in the order of experimental procedures, beginning with the assignment of diffraction rings to lattice planes based on measured ring diameters and corresponding Bragg angles.

The NaCl sample (Figure 3a) displays a well-defined series of concentric rings with variable intensity. Up to seven rings are distinguishable, with higher intensities noted for the second and third reflections, consistent with the fcc (face-centered cubic) symmetry. In contrast, the CsCl pattern (Figure 3b) also reveals seven rings, albeit with a different intensity distribution. The first reflection in CsCl is significantly more intense, a feature typical of bcc (body-centered cubic) lattices where the (100) reflection is forbidden and the (110) family dominates the low-angle region.



a) NaCl b) CsCl

Figure 3 – Debye-Scherrer pattern: sample thickness: 0.4 mm; exposure time: 2.5 h

The results of evaluation of the Debye-Scherrer rings of NaCl are summarized in Table 1.

Table 1 – Evaluation of the Debye-Scherrer rings of NaCl. Distance between the sample and film 32 mm + 0.5 mm film thickness. Wavelength: $\lambda(K_\alpha)$ = 71.1 pm.

No.	Intensity	D, mm	Θ, °	$\frac{\sin\theta_n}{\sin\theta_1}$	$\frac{N_n}{N_{min}}$	hkl	d, pm	a, pm
1	very weak	14.7	6.5	1.00	1.00	011	318.9	552.4
2	very strong	16.8	7.1	1.27	1.25	002	283.8	567.3
3	very strong	24.5	10.2	2.64	2.64	022	198.7	562.3
4	strong	30.6	12.7	3.84	3.82	222	163.1	564.3
5	weak	36.1	14.5	5.12	5.12	004	141.2	564.2
6	medium	42.1	16.3	6.41	6.41	024	125.9	563.1
7	weak	47.5	18.2	7.78	7.75	224	114.5	560.5

The data in Table 1 show a systematic increase in ring diameter and diffraction angle with each successive reflection. The $sin^2(\theta)$ ratios correspond well with theoretical predictions for a fcc lattice, particularly when the first reflection is indexed as the (111) plane. The Miller indices are consistent with the fcc selection rules, showing only combinations where the sum of indices is even or odd, but not mixed. The lattice constant calculated from these reflections averages a = 562.0 ± 4.7 pm, which aligns closely with the known literature value of 563.9 pm [13].

The structural analysis was then extended to CsCl using the same Debye-Scherrer method. The results are summarized in Table 2, listing the measured ring diameters, glancing angles, interplanar distances, and assigned lattice planes.

Table 2 – Evaluation of the Debye-Scherrer rings of CsCl. Distance between the sample and film: 30 mm + 0.5 mm film thickness. Wavelength: $\lambda(K_\alpha)$ = 71.1 pm.

No.	Intensity	D, mm	Θ, °	$\frac{\sin\theta_n}{\sin\theta_1}$	$\frac{N_n}{N_{min}}$	hkl	d, pm	a, pm
1	very strong	15.5	7.1	1.00	1.00	011	287.7	407.4
2	very weak	19.4	8.7	-	-	002	206.2	412.3
3	very strong	22.4	10.0	1.94	2.01	002	204.7	409.3
4	strong	27.9	12.4	2.97	3.00	112	166.8	408.6
5	weak	32.8	14.3	3.94	4.00	022	144.8	409.7
6	medium	37.9	15.8	4.92	5.00	013	129.7	410.4
7	weak	42.6	17.7	5.93	6.00	222	118.2	409.5

The progression of ring diameters and corresponding θ-values for both NaCl and CsCl displays a clear and regular increase, as expected from Bragg’s Law for cubic crystal systems. In the case of NaCl, the ratios $\frac{\sin\theta_n}{\sin\theta_1}$ align well with the theoretical sequence 1:1.33:2.66:4.00:5.33:6.66:8.00, which corresponds to the allowed reflections in an fcc lattice. Notably, all observed (hkl) indices follow the selection rule for fcc crystals: only reflections with either all even or all odd indices appear. This, combined with the calculated average lattice parameter of 562.0 ± 4.7 pm, supports the conclusion that NaCl crystallizes in the face-centered cubic structure, in agreement with the standard literature value of 563.9 pm [13], [14].

In contrast, the CsCl results exhibit a different behavior. The reflections do not adhere to the even-only or odd-only selection rules. Instead, the Miller indices include combinations such as (011), (112), and (013), which are characteristic of a bcc lattice, where allowed reflections must satisfy the condition that the sum $h+k+l$ is even. This explains the absence of the (100) reflection and the dominance of other even-summed planes. The $sin^2(\theta)$ quotient progression of 1:2:3:4:5:6 corresponds well to theoretical bcc expectations, and the mean lattice constant of 409.6±1.7pm closely matches the reference value of 411.0 pm [13], [14].

Overall, both data sets demonstrate high internal consistency and conformity with established crystallographic models. The patterns and lattice constants extracted from the Debye-Scherrer rings match not only theoretical expectations but also previously published experimental values [13], [14]. The clarity of the second and third rings in NaCl and the first in CsCl reinforces the known differences in scattering behavior and atomic packing factors between fcc and bcc structures.

4. Conclusions

The structural analysis of powdered NaCl and CsCl using Debye-Scherrer diffraction successfully confirmed their respective crystal systems: NaCl adopts a fcc lattice, while CsCl forms a bcc lattice.

For NaCl, seven diffraction rings were clearly observed, with $sin^2(\theta)$ ratios following the expected fcc pattern. The calculated average lattice constant was a = 562.0 ± 4.7 pm, consistent with the known value of 563.9 pm.

CsCl exhibited a characteristic bcc reflection pattern with even-summed Miller indices, yielding a mean lattice constant of a = 409.6 ± 1.7 pm, matching the standard 411.0 pm.

The number of atoms per unit cell was estimated as ~4 for NaCl and ~2 for CsCl, confirming their fcc and bcc identities based on density and unit cell volume calculations.

A major trend observed was the alignment of the $\sin^2(\theta)$ quotient progressions with theoretical expectations for each lattice type, validating the indexing and structure determination methodology.

The study successfully addressed the research objective by demonstrating that ring pattern analysis and basic geometric measurements can be used to identify crystal systems, calculate lattice constants, and confirm unit cell content in cubic ionic crystals.

These findings can support educational laboratory training in crystallography and may serve as a reference protocol for rapid phase identification in powder samples using basic X-ray techniques.

A constraint of the study lies in the limited exposure times, which affected the visibility of weaker high-order reflections. Future studies may incorporate synchrotron sources or longer exposures for improved resolution of marginal rings.

References

[1] K. Wang, W. Yang, X. Gao, and X. Wang, “Effect of preparation conditions on crystal size and pore structure of pseudoboehmite,” Tianjin Daxue Xuebao (Ziran Kexue yu Gongcheng Jishu Ban)/Journal Tianjin Univ. Sci. Technol., vol. 46, no. 10, pp. 934–938, Oct. 2013, doi: 10.11784/TDXB20131013.

[2] K. C. Ross, J. A. Petrus, and A. M. McDonald, “An empirical assessment of two-dimensional (2D) Debye-Scherrer-type image-plate X-ray diffraction data collapsed into a 1D diffractogram,” Powder Diffr., vol. 29, no. 4, pp. 337–345, Jun. 2014, doi: 10.1017/S0885715614000487.

[3] H. Yanxon et al., “Artifact identification in X-ray diffraction data using machine learning methods,” J. Synchrotron Radiat., vol. 30, pp. 137–146, Jan. 2023, doi: 10.1107/S1600577522011274.

[4] S. H. Teh and I. I. Yaacob, “Synthesis and characterisation of electrodeposited iron-platinum nanostructured thin films,” Mater. Res. Innov., vol. 13, no. 3, pp. 199–202, Sep. 2009, doi: 10.1179/143307509X437617.

[5] Y. Teshima and T. Matsumoto, “Continuous deformation of sphere-packing structures extending over simple, body-centered, and face-centered cubic lattices,” Glas. Phys. Chem., vol. 38, no. 1, pp. 49–54, Feb. 2012, doi: 10.1134/S1087659612010154.

[6] S. V. Karpenko, A. P. Savintsev, and A. I. Temrokov, “Anomalous behavior of surface characteristics of certain ionic crystals under high pressures,” Dokl. Phys., vol. 53, no. 3, pp. 128–132, 2008, doi: 10.1134/S102833580803004X.

[7] X. Wang et al., “Revisiting lattice thermal conductivity of CsCl: The crucial role of quartic anharmonicity,” Appl. Phys. Lett., vol. 124, no. 17, Apr. 2024, doi: 10.1063/5.0201393.

[8] H. Ling, J. Montoya, L. Hung, and M. Aykol, “Solving inorganic crystal structures from X-ray powder diffraction using a generative first-principles framework,” Comput. Mater. Sci., vol. 214, p. 111687, Nov. 2022, doi: 10.1016/J.COMMATSCI.2022.111687.

[9] P. Tolédano, K. Knorr, L. Ehm, and W. Depmeier, “Phenomenological theory of the reconstructive phase transition between the NaCl and CsCl structure types,” Phys. Rev. B - Condens. Matter Mater. Phys., vol. 67, no. 14, Apr. 2003, doi: 10.1103/PHYSREVB.67.144106.

[10] Y. Liu et al., “Progress and challenges in structural, in situ and operando characterization of single-atom catalysts by X-ray based synchrotron radiation techniques,” Chem. Soc. Rev., Oct. 2024, doi: 10.1039/D3CS00967J.

[11] J. Santana-Andreo, H. D. Saßnick, and C. Cocchi, “Thermodynamic stability and vibrational properties of multi-alkali antimonides,” JPhys Mater., vol. 7, no. 3, Jul. 2024, doi: 10.1088/2515-7639/AD510B.

[12] F. Logiurato, L. M. Gratton, and S. Oss, “Optical Simulation of Debye-Scherrer Crystal Diffraction,” Phys. Teach., vol. 46, no. 2, pp. 109–112, Feb. 2008, doi: 10.1119/1.2834534.

[13] “Introduction to Solid State Physics Charles Kittel,” 2005.

[14] D. W. Snoke, “Solid State Physics: Essential Concepts,” Jan. 2020, doi: 10.1017/9781108123815.

Information about authors:

Marry Brewer – PhD, Research Assistant, Department of Materials Science, Sheffield Hallam University, Sheffield, United Kingdom, b248967468@gmail.com

Almas Sheriyev – MSc, Research Assistant, Department of Materials Science, Sheffield Hallam University, Sheffield, United Kingdom, sheriyev.almas@gmail.com

Author Contributions:

Marry Brewer – concept, methodology, interpretation, drafting, editing, funding acquisition.

Almas Sheriyev – resources, data collection, testing, modeling, analysis, visualization.

Conflict of Interest: The authors declare no conflict of interest.

Use of Artificial Intelligence (AI): The authors declare that AI was not used.

Received: 11.05.2025
Revised: 03.06.2025
Accepted: 07.06.2025
Published: 11.06.2025